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# फेरोक्रोमियम का रासायनिक विश्लेषण

## भाग 3 अल्प कार्बन फेरोक्रोमियम में स्पेक्ट्रमी प्रकाशमिति पद्धति द्वारा फॉस्फोरस ज्ञात करना

*Indian Standard*

# CHEMICAL ANALYSIS OF FERROCHROMIUM

## PART 3 DETERMINATION OF PHOSPHORUS IN LOW CARBON FERROCHROMIUM BY SPECTROPHOTOMETRIC METHOD

UDC 669·15·26 : 543·42 [ 549·22 ]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

*September* 1992 **Price Group 1**

Methods of Chemical Analysis of Ferrous Metals Sectional Committee, MTD 2

## FOREWORD

This Indian Standard ( Part 3 ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1559 : 1961 'Methods of chemical analysis of ferroalloys' covers the chemical analysis of different ferroalloys, namely, ferrosilicon, ferrochromium, ferromanganese and speigeleisen, silicomanganese, ferromolybdenum, ferrophosphorus, ferrotitanium, ferrovanadium and ferrotungsten. While reviewing this standard, the Sectional Committee had decided that a separate series for each ferroalloy be prepared. Accordingly, IS 1559 in its various parts was published for the chemical analysis of ferrosilicon.

The chemical analysis of ferromolybdenum is covered in a series of parts of IS 12614. The chemical analysis of ferrochromium is covered in a series of parts of this standard. With the publication of these parts the analysis of relevant constituent prescribed for ferrochromium in IS 1559 : 1961 will be superseded. This part covers the method for determination of phosphorus in low carbon ferrochromium. The other parts are:

Part 1 Determination of silicon in high carbon ferrochromium
Part 2 Determination of silicon in low carbon ferrochromium
Part 4 Determination of total sulphur in low and high carbon ferrochromium
Part 5 Determination of chromium in high carbon ferrochromium
Part 6 Determination of chromium in low carbon ferrochromium
Part 7 Determination of nitrogen in high and low carbon ferrochromium
Part 8 Determination of carbon ( total ) in low and high carbon ferrochromium

In this revision the 'Alkalimetric Method' for determination of phosphorus in ferrochromium has been replaced by 'Spectrophotometric Method'.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( *revised* )'.

## *Indian Standard*

# CHEMICAL ANALYSIS OF FERROCHROMIUM

## PART 3 DETERMINATION OF PHOSPHORUS IN LOW CARBON FERROCHROMIUM BY SPECTROPHOTOMETRIC METHOD

### 1 SCOPE

This standard ( Part 3 ) describes the method for determination of phosphorus in low carbon ferrochromium ( containing carbon up to one percent ).

### 2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard :

| *IS No.* | *Title* |
|---|---|
| 264 : 1976 | Nitric acid ( *second revision* ) |
| 265 : 1987 | Hydrochloric acid ( *third revision* ) |
| 1070 : 1992 | Reagent grade water — Specification ( *third revision* ) |
| 1472 : 1977 | Methods of sampling of ferroalloys for determination of chemical composition ( *second revision* ) |

### 3 SAMPLING

The sample shall be drawn and prepared in accordance with IS 1472 : 1977.

### 4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water ( *see* IS 1070 : 1992 ) shall be employed for the test.

### 5 DETERMINATION OF PHOSPHORUS

#### 5.1 Outline of the Method

Sample is dissolved in hydrochloric acid, nitric acid and perchloric acid, when phosphorus is oxidized to orthophosphoric acid. Fumed with perchloric acid, after addition of sodium chloride to remove chromium as chromylchoride. The phosphorus in the solution is reacted with ammonium molybdate and reduced with hydrazine sulphate solution to give molybdenum blue. The intensity of the colour is measured spectrophotometrically at 820 nm.

#### 5.2 Reagents

**5.2.1** *Hydrochloric Acid*, rd = 1·16 ( conforming to IS 265 : 1987 ).

**5.2.2** *Dilute Hydrochloric Acid*, 35 percent ( *v/v* ).

**5.2.3** *Nitric Acid*, rd = 1·42 ( conforming to IS 264 : 1976 ).

**5.2.4** *Perchloric Acid*, 60 percent.

**5.2.5** *Dilute Sulphuric Acid*, 1 : 4, 3 : 5 ( *v/v* ).

**5.2.6** *Aqua Ammonium*

**5.2.7** *Sodium Chloride*, solid.

**5.2.8** *Sodium Sulphite* ( $Na_2SO_3$ ) *Solution*, 10 percent ( *m/v* ) should be freshly prepared.

**5.2.9** *Ammonium Molybdate Solution* ( *Solution A* )

Add 20 g of ammonium molybdate [ $(NH_4)_6 Mo_7O_{24}.H_2O$ ) ] to 100 ml of warm water. Add 800 ml of dilute sulphuric acid ( 3 : 5 ) and cool to room temperature. Make up the volume to 1 litre with water.

**5.2.10** *Hydrazine Sulphate Solution* ( *Solution B* )

Dissolve 1·5 g of hydrazine sulphate ( $N_2H_4 \cdot H_2SO_4$ ) in 1 litre water.

**5.2.11** *Phosphorus Standard Solution* — ( 1 ml = 0·1 mg P ).

Weigh accurately 2·292 g of anhydrous $Na_2HPO_4$ previously dried at 105°C. Transfer to 500 ml volumetric flask. Dissolve in 100 ml of water, dilute to mark and mix. Pipette out 10 ml of solution and dilute to 100 ml with water.

**5.2.12** *Colour Development Solution*

Mix 25 ml of solution (*A*) and 10 ml of solution (*B*) and make up the volume to 100 ml with water. This should be prepared just before use.

#### 5.3 Procedure

**5.3.1** Weigh 0·500 g of sample and transfer to 250 ml beaker. Wet the sample with 10 ml of water. Add 20 ml hydrochloric acid, 5 ml nitric acid and 25 ml perchloric acid and reflux for 15 minutes after fuming starts. Keep it on the hot plate for dissolution for about half-an-hour, till the mass in the beaker becomes clear.

**5.3.2** Add dilute hydrochloric acid ( 35 percent ) dropwise, till the solution turns green. Keep on a hot plate and reflux for 5 minutes.

**5.3.3** Repeat the operation ( **5.3.2** ), 3 or 4 times and finally add a pinch of sodium chloride and reflux till all the chromium is expelled.

**5.3.4** Cool solution ( **5.3.3** ) and add about 40 ml of water to dissolve the soluble salts and make up the volume to 100 ml into a volumetric flask. Filter the solution using medium textured filter paper and collect the filtrate in a 250 ml beaker.

**5.3.5** Pipette out 10 ml of filtrate ( **5.3.4** ) into a 250 ml beaker. Add 10 ml of sodium sulphite solution ( freshly prepared ) and 10 ml of water. Boil for 1 minute.

**5.3.6** Add 25 ml of colour development solution ( **5.2.12** ) and immerse the beaker in a boiling water bath and keep it for 10 minutes for full colour development. Cool to room temperature and make up the volume to 100 ml with distilled water in volumetric flask.

**5.3.7** Transfer a suitable portion of **5.3.6** to 1 cm cell and adjust the spectrophotometer at 820 nm and measure the absorbance against the reagent blank.

**5.3.8** *Blank*

Carry out a blank determination using same amount of reagents and chemicals and following the procedure specified in **5.3.1** to **5.3.7**.

**5.3.9** *Preparation of the Calibration Curve*

Transfer 0, 1, 2, 3, 4 and 5 ml of standard phosphorus solution ( 1 ml = 0·1 mg P ) ( *see* **5.2.11** ) 3, P to 500 ml beaker and proceed according to **5.3.1** to **5.3.7**. The flask containing the zero ml of phosphorus solution is regarded as blank and the net value of spectrophotometric reading is plotted against mg of phosphorus.

**5.3.10** Convert the spectrophotometric readings of test solution to mg of phosphorus obtained from calibration curve and calculate the percentage of phosphorus.

**5.3.11** *Calculation*

$$\text{Phosphorus, percent by mass} = \frac{A}{B} \times \frac{1}{10}$$

where

$A$ = mass in mg, of phosphorus found in solution; and

$B$ = mass in g, of the sample taken.

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